

4/2003 SN 09/806,442

FAST - [Default FAST Workspace (Flat Panel LANDS) [FAST] wsp1]

File View Edit Tools Window Help



Drafts

Pending

Active

- L1: (237) (205/266-268).CCLs.
- L2: (32763) sulfite or sulfites
- L3: (37976) soSub.3
- L4: (35820) "so.sub.3"
- L5: (62544) 12 or 14
- L6: (40) 11 and 15
- L7: (4001588) gold or Au
- L8: (40) 16 and 17
- L9: (516) (204/272).CCLs.
- L10: (534470) inner same outer
- L11: (95919) anode or anodes
- L12: (3445) 110 same 111
- L13: (103) 19 and 112
- L14: (127101) shield or shields
- L15: (2296) 114 same 111
- L16: (7) 113 and 115

- Failed
- Saved
- Favorites
- Tagged (0)
- UDC
- Queue
- Trash

USPAT

Default operator: OR

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113 and 115

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U	I	PT	P	Document ID	Issue Date	Pages	Title	Current OR	Current XRef Retrieval C	Inventor	S	C	2	3
				US 6258251 B1	20010710	11	Electrolytic cell	205/771	204/272;	Gowans, Bruce E. et al.				
				US 5865965 A	19990202	45	Apparatus for electrochemical	204/268	204/272;	Enda, Masami et al.				
				US 5650053 A	19970722	13	Electrorefining cell with parallel	204/212	204/215;	Gay, Eddie C. et al.				
				US 4352727 A	19821005	6	Rotogravure cylinder plating	204/212	204/227;	Metzger, Hubert				
				US 4331527 A	19820525	6	and de-plating apparatus	204/272;	204/272;	Metzger, Hubert				
				US 3951774 A	19760420	5	Rotogravure cylinder plating apparatus	204/212	204/285;	Jones, Glenn C.				
				US 3929592 A	19751230	7	Electrolytic cell construction	204/272	205/452	Klingenmaier, Otto J.				
							Plating apparatus and method for rotary engine housings	205/122	204/237;					
									204/272;					

Ready

Ready

Document ID	Pages	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	251	252	253	254	255	256	257	258	259	260	261	262	263	264	265	266	267	268	269	270	271	272	273	274	275	276	277	278	279	280	281	282	283	284	285	286	287	288	289	290	291	292	293	294	295	296	297	298	299	300	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334	335	336	337	338	339	340	341	342	343	344	345	346	347	348	349	350	351	352	353	354	355	356	357	358	359	360	361	362	363	364	365	366	367	368	369	370	371	372	373	374	375	376	377	378	379	380	381	382	383	384	385	386	387	388	389	390	391	392	393	394	395	396	397	398	399	400	401	402	403	404	405	406	407	408	409	410	411	412	413	414	415	416	417	418	419	420	421	422	423	424	425	426	427	428	429	430	431	432	433	434	435	436	437	438	439	440	441	442	443	444	445	446	447	448	449	450	451	452	453	454	455	456	457	458	459	460	461	462	463	464	465	466	467	468	469	470	471	472	473	474	475	476	477	478	479	480	481	482	483	484	485	486	487	488	489	490	491	492	493	494	495	496	497	498	499	500	501	502	503	504	505	506	507	508	509	510	511	512	513	514	515	516	517	518	519	520	521	522	523	524	525	526	527	528	529	530	531	532	533	534	535	536	537	538	539	540	541	542	543	544	545	546	547	548	549	550	551	552	553	554	555	556	557	558	559	560	561	562	563	564	565	566	567	568	569	570	571	572	573	574	575	576	577	578	579	580	581	582	583	584	585	586	587	588	589	590	591	592	593	594	595	596	597	598	599	600	601	602	603	604	605	606	607	608	609	610	611	612	613	614	615	616	617	618	619	620	621	622	623	624	625	626	627	628	629	630	631	632	633	634	635	636	637	638	639	640	641	642	643	644	645	646	647	648	649	650	651	652	653	654	655	656	657	658	659	660	661	662	663	664	665	666	667	668	669	670	671	672	673	674	675	676	677	678	679	680	681	682	683	684	685	686	687	688	689	690	691	692	693	694	695	696	697	698	699	700	701	702	703	704	705	706	707	708	709	710	711	712	713	714	715	716	717	718	719	720	721	722	723	724	725	726	727	728	729	730	731	732	733	734	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760	761	762	763	764	765	766	767	768	769	770	771	772	773	774	775	776	777	778	779	780	781	782	783	784	785	786	787	788	789	790	791	792	793	794	795	796	797	798	799	800	801	802	803	804	805	806	807	808	809	810	811	812	813	814	815	816	817	818	819	820	821	822	823	824	825	826	827	828	829	830	831	832	833	834	835	836	837	838	839	840	841	842	843	844	845	846	847	848	849	850	851	852	853	854	855	856	857	858	859	860	861	862	863	864	865	866	867	868	869	870	871	872	873	874	875	876	877	878	879	880	881	882	883	884	885	886	887	888	889	890	891	892	893	894	895	896	897	898	899	900	901	902	903	904	905	906	907	908	909	910	911	912	913	914	915	916	917	918	919	920	921	922	923	924	925	926	927	928	929	930	931	932	933	934	935	936	937	938	939	940	941	942	943	944	945	946	947	948	949	950	951	952	953	954	955	956	957	958	959	960	961	962	963	964	965	966	967	968	969	970	971	972	973	974	975	976	977	978	979	980	981	982	983	984	985	986	987	988	989	990	991	992	993	994	995	996	997	998	999	1000	1001	1002	1003	1004	1005	1006	1007	1008	1009	1010	1011	1012	1013	1014	1015	1016	1017	1018	1019	1020	1021	1022	1023	1024	1025	1026	1027	1028	1029	1030	1031	1032	1033	1034	1035	1036	1037	1038	1039	1040	1041	1042	1043	1044	1045	1046	1047	1048	1049	1050	1051	1052	1053	1054	1055	1056	1057	1058	1059	1060	1061	1062	1063	1064	1065	1066	1067	1068	1069	1070	1071	1072	1073	1074	1075	1076	1077	1078	1079	1080	1081	1082	1083	1084	1085	1086	1087	1088	1089	1090	1091	1092	1093	1094	1095	1096	1097	1098	1099	1100	1101	1102	1103	1104	1105	1106	1107	1108	1109	1110	1111	1112	1113	1114	1115	1116	1117	1118	1119	1120	1121	1122	1123	1124	1125	1126	1127	1128	1129	1130	1131	1132	1133	1134	1135	1136	1137	1138	1139	1140	1141	1142	1143	1144	1145	1146	1147	1148	1149	1150	1151	1152	1153	1154	1155	1156	1157	1158	1159	1160	1161	1162	1163	1164	1165	1166	1167	1168	1169	1170	1171	1172	1173	1174	1175	1176	1177	1178	1179	1180	1181	1182	1183	1184	1185	1186	1187	1188	1189	1190	1191	1192	1193	1194	1195	1196	1197	1198	1199	1200	1201	1202	1203	1204	1205	1206	1207	1208	1209	1210	1211	1212	1213	1214	1215	1216	1217	1218	1219	122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Document ID #	Pages	1	2	3	U	S	C	P	Kind Codes	Source
US 6126797 A	40									USPAT
US 6117285 A	32									USPAT
US 6103078 A	19									USPAT
US 6007686 A	23									USPAT
US 5972180 A	8									USPAT
US 5882502 A	22									USPAT
US 5873966 A	9									USPAT

US-PAT-NO: 5882502

DOCUMENT-IDENTIFIER: US 5882502 A

TITLE: Electrochemical system and method

----- RWIC -----

Detailed Description Text - DETX (14):

The cell comprises an annular body in which the outer surface 40 provides the cathode or anode wall and the inner wall 41 provides the respective solution electrode. It is possible to add another annular ring (not shown) to act as another cathode or anode, further increasing the capacity of the cell. Tangential inlets 42 and outlets 43 may be provided to give turbulent flow and good mixing in the cells. A non-conductive lid 44 is used in the cell and electrical connections are provided to the anode/cathode and the solution electrode.

Claims Text - CCTX (12):

7. An electrochemical metal recovery apparatus as in claim 1 wherein either the anode cell or the cathode cell or both the anode cell and cathode cell comprises an annular body having inner and outer cylindrical walls defining the annular body therebetween, the inner cylindrical wall being the anode or cathode respectively and the outer cylindrical wall being the respective solution electrode, and wherein a tangential electrolyte inlet is provided at one axial end of the annular body and a tangential electrolyte outlet at the other axial end of the annular body.

Current US Cross Reference Classification - CCCR (8):

201/272

U.S. Patent

Mar. 16, 1999

Sheet 4 of 13

5,882,502

FIG. 4B

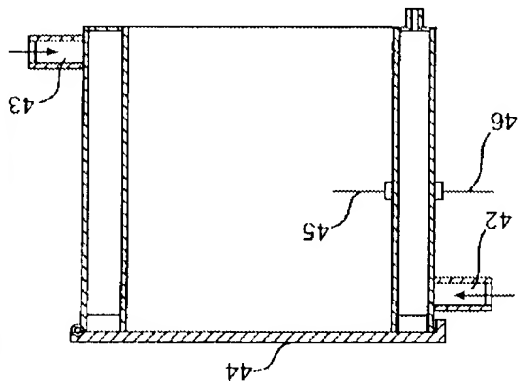
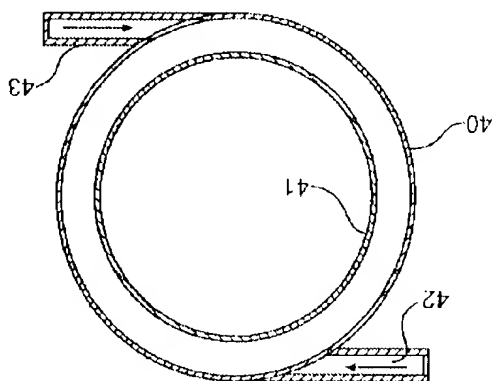


FIG. 4A



Document ID	Pages	1	2	3	U	S	C	P	Kind Code	Sub
US 6258251 B1	11								USPAT	
US 5865965 A	45								USPAT	
US 5650053 A	13								USPAT	
US 4352727 A	6								USPAT	
US 4331527 A	6								USPAT	
US 3951774 A	5								USPAT	
US 3929592 A	7								USPAT	

US-PAT-NO: 5865965

DOCUMENT-IDENTIFIER: US 5865965 A

TITLE: Apparatus for electrochemical decontamination of radioactive metallic waste

----- KWIC -----

Abstract Text - ABTX (1):

An insulating shield plate (1) divides a room of an electrolysis bath (2) into an anodic chamber (13) and a cathodic chamber (14). An anode (5) is disposed in the anodic chamber (13), and a cathode (6), metallic waste (7) and heater (4) are disposed in the cathodic chamber (14). An electrolyte (3) stored in the electrolysis bath (2) flows in a circulation path from a circulating pump (10) through a circulating line (12) and filter (11) to return the electrolysis bath (2). An exhaust gas processing device 9 is connected to the electrolysis bath (2). When a voltage is supplied to the anode (5) and cathode (6), a surface of the metallic waste (7) is charged in a positive polarity to dissolve a base metal of the metallic waste (7). Further, a cylindrical anode (33) is arranged in an electrolyte (32) in an electrolysis bath (31), and a cylindrical metal (34) as a metallic waste is arranged in the cylindrical anode (33). After a bar-shaped cathode (35) is arranged in the cylindrical metal (34), a DC voltage is supplied to the cylindrical anode (33) and the bar-shaped cathode (35). At this time, the system may be constructed in that oxygen occurs by charging in a positive polarity in an inner surface of the cylindrical metal (34) and the base metal of the cylindrical shape is dissolved.

Brief Summary Text - BSTD (19):

an insulating shield plate for dividing a room of the electrolysis bath into an anodic chamber and a cathodic chamber, and the insulating shield plate which is set in a U-shape along three inner wall surfaces of the electrolysis bath.

Brief Summary Text - BSTD (33):

any one of electrodes of two kinds of the anode and the cathode for electrolysis has a cylindrical shape along the inner shape of the metallic waste having a curved portion, and the other of the electrodes is formed in a bar-shaped in correspondence with an inner shape of the metallic waste.

Brief Summary Text - BSTD (43):

a cylindrical anode which is arranged on an inner wall of the cylindrical insulating shield body;

Brief Summary Text - BSTD (53):

an anode which is arranged at the bottom of the insulating shield vessel;

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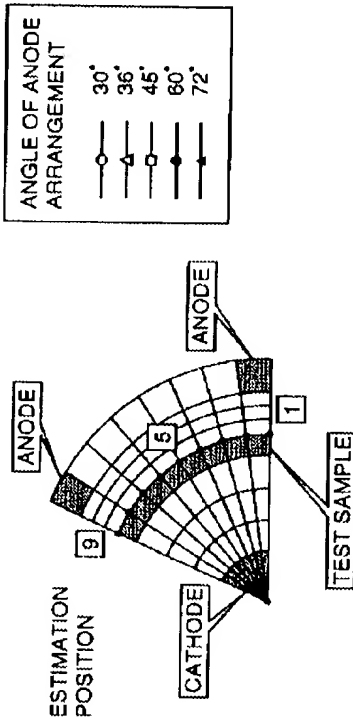


FIG. 31A

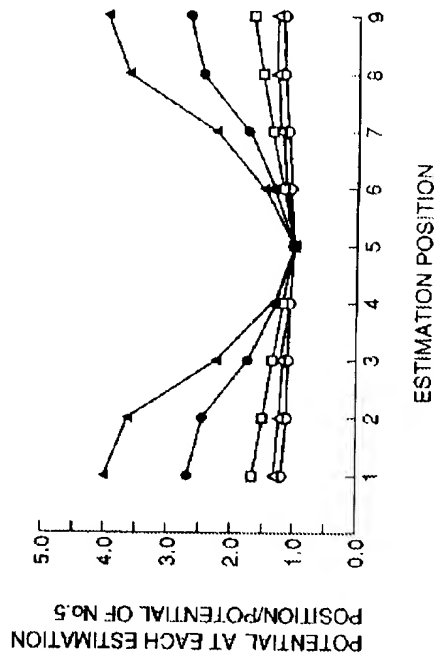


FIG. 31B

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ID	Pages	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	251	252	253	254	255	256	257	258	259	260	261	262	263	264	265	266	267	268	269	270	271	272	273	274	275	276	277	278	279	280	281	282	283	284	285	286	287	288	289	290	291	292	293	294	295	296	297	298	299	300	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334	335	336	337	338	339	340	341	342	343	344	345	346	347	348	349	350	351	352	353	354	355	356	357	358	359	360	361	362	363	364	365	366	367	368	369	370	371	372	373	374	375	376	377	378	379	380	381	382	383	384	385	386	387	388	389	390	391	392	393	394	395	396	397	398	399	400	401	402	403	404	405	406	407	408	409	410	411	412	413	414	415	416	417	418	419	420	421	422	423	424	425	426	427	428	429	430	431	432	433	434	435	436	437	438	439	440	441	442	443	444	445	446	447	448	449	450	451	452	453	454	455	456	457	458	459	460	461	462	463	464	465	466	467	468	469	470	471	472	473	474	475	476	477	478	479	480	481	482	483	484	485	486	487	488	489	490	491	492	493	494	495	496	497	498	499	500	501	502	503	504	505	506	507	508	509	510	511	512	513	514	515	516	517	518	519	520	521	522	523	524	525	526	527	528	529	530	531	532	533	534	535	536	537	538	539	540	541	542	543	544	545	546	547	548	549	550	551	552	553	554	555	556	557	558	559	560	561	562	563	564	565	566	567	568	569	570	571	572	573	574	575	576	577	578	579	580	581	582	583	584	585	586	587	588	589	590	591	592	593	594	595	596	597	598	599	600	601	602	603	604	605	606	607	608	609	610	611	612	613	614	615	616	617	618	619	620	621	622	623	624	625	626	627	628	629	630	631	632	633	634	635	636	637	638	639	640	641	642	643	644	645	646	647	648	649	650	651	652	653	654	655	656	657	658	659	660	661	662	663	664	665	666	667	668	669	670	671	672	673	674	675	676	677	678	679	680	681	682	683	684	685	686	687	688	689	690	691	692	693	694	695	696	697	698	699	700	701	702	703	704	705	706	707	708	709	710	711	712	713	714	715	716	717	718	719	720	721	722	723	724	725	726	727	728	729	730	731	732	733	734	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760	761	762	763	764	765	766	767	768	769	770	771	772	773	774	775	776	777	778	779	780	781	782	783	784	785	786	787	788	789	790	791	792	793	794	795	796	797	798	799	800	801	802	803	804	805	806	807	808	809	810	811	812	813	814	815	816	817	818	819	820	821	822	823	824	825	826	827	828	829	830	831	832	833	834	835	836	837	838	839	840	841	842	843	844	845	846	847	848	849	850	851	852	853	854	855	856	857	858	859	860	861	862	863	864	865	866	867	868	869	870	871	872	873	874	875	876	877	878	879	880	881	882	883	884	885	886	887	888	889	890	891	892	893	894	895	896	897	898	899	900	901	902	903	904	905	906	907	908	909	910	911	912	913	914	915	916	917	918	919	920	921	922	923	924	925	926	927	928	929	930	931	932	933	934	935	936	937	938	939	940	941	942	943	944	945	946	947	948	949	950	951	952	953	954	955	956	957	958	959	960	961	962	963	964	965	966	967	968	969	970	971	972	973	974	975	976	977	978	979	980	981	982	983	984	985	986	987	988	989	990	991	992	993	994	995	996	997	998	999	1000	1001	1002	1003	1004	1005	1006	1007	1008	1009	1010	1011	1012	1013	1014	1015	1016	1017	1018	1019	1020	1021	1022	1023	1024	1025	1026	1027	1028	1029	1030	1031	1032	1033	1034	1035	1036	1037	1038	1039	1040	1041	1042	1043	1044	1045	1046	1047	1048	1049	1050	1051	1052	1053	1054	1055	1056	1057	1058	1059	1060	1061	1062	1063	1064	1065	1066	1067	1068	1069	1070	1071	1072	1073	1074	1075	1076	1077	1078	1079	1080	1081	1082	1083	1084	1085	1086	1087	1088	1089	1090	1091	1092	1093	1094	1095	1096	1097	1098	1099	1100	1101	1102	1103	1104	1105	1106	1107	1108	1109	1110	1111	1112	1113	1114	1115	1116	1117	1118	1119	1120	1121	1122	1123	1124	1125	1126	1127	1128	1129	1130	1131	1132	1133	1134	1135	1136	1137	1138	1139	1140	1141	1142	1143	1144	1145	1146	1147	1148	1149	1150	1151	1152	1153	1154	1155	1156	1157	1158	1159	1160	1161	1162	1163	1164	1165	1166	1167	1168	1169	1170	1171	1172	1173	1174	1175	1176	1177	1178	1179	1180	1181	1182	1183	1184	1185	1186	1187	1188	1189	1190	1191	1192	1193	1194	1195	1196	1197	1198	1199	1200	1201	1202	1203	1204	1205	1206	1207	1208	1209	1210	1211	1212	1213	1214	1215	1216	1217	1218	1219	122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US-PAT-NO: 5650053

DOCUMENT-IDENTIFIER: US 5650053 A

TITLE: Electrorefining cell with parallel electrode/concentric cylinder cathode

----- RWIC -----

Abstract Text - ABTX (1):

A cathode/anode arrangement for use in an electrolytic cell is adapted for electrochemically refining spent nuclear fuel from a nuclear reactor and recovering purified uranium for further treatment and possible recycling as a fresh blanket or core fuel in a nuclear reactor. The arrangement includes a plurality of anodic dissolution baskets that are each attached to a respective support rod, are submerged in a molten lithium halide salt, and are rotationally displaced. An inner hollow cylindrical-shaped cathode is concentrically disposed about the anodic dissolution baskets. A plurality of anodic dissolution baskets, while an outer hollow cylindrical-shaped cathode is disposed about the anodic dissolution baskets. Uranium is transported from the anodic baskets and deposited in a uniform cylindrical shape on the inner and outer cathode cylinders by rotating the anodic baskets within the molten lithium halide salt. Scrapers located in each anodic basket scrape and remove the spent fuel deposits on the surfaces of the inner and outer cathode cylinders, with the spent fuel falling to the bottom of the cell for removal. Cell resistance is reduced and uranium deposition rate enhanced by increasing the electrode area and reducing the anode-cathode spacing. Collection efficiency is enhanced by trapping and recovery of uranium dendrites scrapped off of the cylindrical cathodes which may be greater in number than two.

Detailed Description Text - DETX (4):

In accordance with the present invention, concentrically disposed within the inner cathode cylinder 14 is an inner cathode cylinder 12. Each of the inner and outer cathode cylinders 14, 12 is maintained in a fixed position relative to each other and is preferably comprised of a suitable metal which may be a ferrous or a molybdenum alloy, as well known in the art. Disposed intermediate the inner cathode cylinder 14 and the inner cathode cylinder 12 and arranged in a spaced manner about the inner cathode cylinder 12 are a plurality of anodic dissolution baskets 16a-18h. Each of the anodic dissolution baskets 16a-18h is attached to and supported by a respective support rod 18. Each of the anodic dissolution baskets 16a-18h is disposed within the inner cathode cylinder 12. Each of the anodic dissolution baskets 16a-18h is similarly coupled to and supported by a respective support rod 18 within the inner cathode cylinder 12. Each of the aforementioned anodic dissolution baskets 16a-18h is in the form of an apertured, porous, container holding a spent nuclear fuel which has been fragmented.

Detailed Description Text - DETX (5):

Also disposed within the inner and outer cathode cylinders 12, 14 is an

United States Patent

Gay et al.

[19]

[11] Patent Number:

5,650,053

[45] Date of Patent:

Jul. 22, 1997

[54] ELECTROREFINING CELL WITH PARALLEL ELECTRODE/CONCENTRIC CYLINDER CATHODE

[75] Inventors: Eddie C. Gay, Park Forest; William E. Miller, Naperville; James J. Laidler, Burr Ridge, all of Ill.

[73] Assignee: The United States of America as represented by the United States Department of Energy, Washington, D.C.

[21] Appl. No.: 562,612

[22] Filed: Nov. 24, 1995

[51] Int. Cl.⁴ C25D 17/00; C25C 3/00; A62D 3/00

[52] U.S. Cl. 205/47; 205/49; 204/272; 204/669; 204/671; 204/243 R; 204/215; 204/277; 204/244; 204/287; 588/201; 52/93

[58] Field of Search 588/201; 976/DIG. 379; 976/DIG. 380; DIG. 286; DIG. 268; 205/44; 46, 47, 49; 204/272; 669, 671, 212, 222; 243 R; 199, 200, 215, 216, 217, 227, 244; 237, 44, 46, 47, 49

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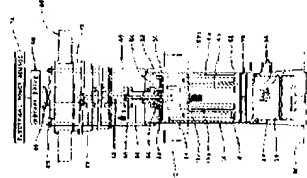
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[57]

ABSTRACT

A cathode-anode arrangement for use in an electrolytic cell is adapted for electrochemically refining spent nuclear fuel from a nuclear reactor and recovering purified uranium for further treatment and possible recycling as a fresh blanket or core fuel in a nuclear reactor. The arrangement includes a plurality of inner anodic dissolution baskets that are each attached to a respective support rod, are submerged in a molten lithium halide salt, and are rotationally displaced. An inner hollow cylindrical-shaped cathode is concentrically disposed about the inner anodic dissolution baskets. Concentrically disposed about the inner cathode in a spaced manner are a plurality of outer anodic dissolution baskets, while an outer hollow cylindrical-shaped cathode is disposed about the outer anodic dissolution baskets. Uranium is transported from the anodic baskets and deposited in a uniform cylindrical shape on the inner and outer cathode cylinders by rotating the anodic baskets within the molten lithium halide salt. Scrapers located on each anodic basket scrape and remove the spent fuel deposits on the surfaces of the inner and outer cathode cylinders, with the spent fuel falling to the bottom of the cell for removal. Cell resistance is reduced and uranium deposition rate enhanced by increasing the electrode area and reducing the anode-cathode spacing. Collection efficiency is enhanced by trapping and recovery of uranium dendrites scrapped off of the cylindrical cathodes which may be greater in number than two.

14 Claims, 8 Drawing Sheets



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[illegible]

Brief Summary Text - B9TX (17):

The operating conditions of the present method are not critical. It is possible to work at room temperature and use, for instance, a concentrated HAUCL₃ sub. 4 solution (about 80-100 g/l of gold) to which concentrated aqueous ammonia (about 30%) is added slowly. Practice has however shown that it is advantageous to use a very concentrated HAUCL₃ sub. 4 solution (more than 500 g/l) with very little free HCl. After adding an excess of ammonia until the precipitation is complete, the precipitate is allowed to settle for decantation. Then the supernatant liquid is removed by syphoning and replaced by pure water. The precipitate is agitated, decantation is carried out again and this washing step is repeated 2 or 3 times until practically all chloride has been eliminated. Washing by filtration should preferably be avoided for the reason of drying up of the precipitate (explosion hazard). Then, after eliminating finely, as well as possible, the rinsing waters, an aqueous solution of ammonium sulfate (commercial grade is suitable) is added for dissolving the precipitate. The quantity and concentration of the sulfate solution is calculated to ensure that the finished ammonium sulfate solution will contain about 100 g/l of gold for about 160-210 g/l of total ammonium sulfate. These values are, in fact, not at all critical but it is preferable to keep as close as possible thereto for convenience when using, thereafter, the obtained ammonium sulfate solution for preparing or maintaining gold plating baths.

Brief Summary Text - B9TX (18):

The aqueous electrolytic bath containing the present ammonium-gold-sulfate complex which is prepared from the above discussed solution is stable and is operated within the 6-8 pH range. It comprises 1 to 50 g/l of gold metal, 10 to 400 g/l of buffering and conducting salts and 10-100 g/l of ammonium sulfate. It may or not further comprise alloying metals in the form of water-soluble compounds or complexes.

Brief Summary Text - BSTX (19):

Thus, this bath which contains the gold in the form of an ammonium gold-sulfate complex is able to provide electrodeposits in acidic media as well as alloyed gold and pure gold, and without needing depolarizers such as arsenic or selenium, even at current densities of 0.25 to 0.5 A/cm², or stabilizers such as polyamines, which was practically impossible with alkali gold-sulfate baths of the prior art. Under such conditions, it is possible with the present bath to obtain bright and semi-bright 24 karat electrodeposits, homogeneous and non-porous. Further, because of the absence of arsenic, this bath is acceptable in the countries in which the use of this element in industry is restricted or prohibited by law.

Brief Summary Text - BSTX (20):

Moreover, this bath being adaptable to acid pH, it is perfectly suited to the desorption at lower rates 2000 mg/l. min. alloys, e.g., 15 to 12 karats. As alloying metals for instance Cd, Pb, Cu, Zn, Al, Sn, Pb, Bi, Ru, etc., can be mentioned with no limitation. The concentration of these metals in the bath can be e.g. 0.01-150 g/l. These metals are present in the bath as their salts, or for complexes conventionally used in gold baths, e.g., the sulfates, nitrates, chlorides, borates, etc.

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obtain lower baral gold-alloys deposits. Similarly, Swiss Pat. No. 534,215 discloses an alkali-sulfite gold bath (sodium or potassium) for the electrolytic deposition of gold-palladium alloys in which there is added, for stabilizing at pH slightly below 7, aliphatic, cycloaliphatic and heterocyclic amines.

However, gold baths prepared in the above described manner have the following disadvantages: during electroplating the bath, the stabilizing anions which enable the latter to operate at pH below 7 are progressively liberated as gold is removed for plating from the complex and as the bath is replenished with gold and stabilizer. Further, these anions are oxidized and decomposed at the anode and, either themselves, or their degradation products impede the operation of the bath. For example by complexing and blocking the common metal ions which must co-deposit with the gold in the plated alloy and, thus, decreasing the useful concentration thereof at the cathode. Consequently, these baths have a very short working life and must be frequently renewed.

It should be moreover remarked that in U.S. Pat. No. 3,787,483, it is mentioned that polyamine A can also consist of ammonia. However, the present Applicant has not succeeded in preparing such a compound of Au^{3+} -wherein A represents NH_3 and, simultaneously, ME represents NH_4^+ . Thus, in contrast to what happens with amines, when ammonium hydroxide is added to a solution of trivalent gold chloride, the mixture precipitates before the introduction of the sulfite. This precipitate, the nature of which is ill-determined, is so-called "fuming gold" in the chemical literature (see the heretofore mentioned reference PASCAL, p. 785). It is interesting to note that the method of the invention is precisely based on this precipitation phenomenon and provides an ammonium-gold-sulfite complex solution which permits, by means of the gold bath prepared therefrom, one to remedy most of the above mentioned disadvantages.

Thus, the method of the invention for the preparation of an aqueous solution of a sulfate complex of monovalent gold and ammonium comprises treating an auriferous solution with a solution of a monovalent ammoniochloric acid (NH₄Cl) aqueous solution with ammoniochloric acid (NH₄Cl) aseptically of a gold compound, then decanting the supernatant solution and washing the residue with a solution of ammoniochloric acid, then precipitating, by decantation, until practically all the chloride ions are eliminated, then adding to the most precipitate a sufficient quantity of an ammonium sulfate solution for dissolving the precipitate and ensuring the reduction of trivalent gold in monovalent gold.

The complete reduction of trivalent gold into monovalent gold can be easily checked by electrolyzing a sample of the above-mentioned solution. When a yield approaching 100% (120 mg/Amp X min) is reached, the reduction is complete. Thus, at least 3.5 equivalent of sulfite (3.5 moles) of ammonium sulfite per mole of precipitated gold (calculated on the number of moles of starting HAuCl_4) will preferably be used.

This process of preparation of a solution of gold-sulfur complex is particularly economical as compared to older processes. Thus, the precipitation of gold with ammonia is substantially quantitative and the precipitate is easily washable by simple decantation as its particles will settle relatively rapidly at the bottom of the container used. The exact nature of this precipitate has not been investigated. Presumably, it is similar to the "finishing" gold compounds reported by DUMAS, RASCHIG and other workers (see PASCAL, *op. cit.*).

may be in admixture with hydroxide $\text{Au}(\text{OH})_3$. The previously proposed chemical formula and the exact nature of the gold-sulfite complex which is obtainable in solution by the present method has never been determined. It is, however, probable that it is not similar to the products mentioned in the *preparat* (see PASCAL, *op. cit.*, p. 117) since its solution is perfectly stable in water, even at slightly acid pH, in contrast to the reports on the ammonium gold-sulfite of formula $(\text{H}_4\text{N})_2\text{Au}(\text{SO}_3)_2 \cdot \text{H}_2\text{O}$. This stability property was indeed absolutely unexpected and constitutes a surprising factor of the present invention.

The operating conditions of the present method are not critical. It is possible to work at room temperature and use, for instance, a concentrated HAcCl₂ solution (about 80–100 g/l of gold) to which concentrated aqueous ammonia (about 30%) is added slowly. Practice has, however shown that it is advantageous to use a very concentrated HAcCl₂ solution (more than 500 g/l) with only a little free HCl. After adding an excess of ammonia until the precipitation is complete, the precipitate is allowed to settle for decantation. Then the supernatant liquid is removed by syphoning and replaced by pure water. The precipitate is agitated, decantation is carried out and the washing step is repeated 2 or 3 times until practically all chloride has been eliminated. Washing by filtration must preferably be avoided for fear of drying up of the precipitate (explosion hazard). Then, after eliminating finally, as well as possible, the rinsing waters, an aqueous solution of ammonium sulfate (commercial grade is suitable) is added for dissolving of the precipitate. The quantity and concentration of the sulfate solution is calculated to ensure that the finished ammonium-gold-sulfate solution will contain about 100 g/l of gold for about 1.60–2.10 g/l of total ammonium sulfate. These values are, in fact, not at all critical but it is preferable to keep as close as possible thereto for convenience when using, thereafter, the obtained ammonium-gold-sulfate solution for preparing or maintaining a solid-gelling bath.

The aqueous electrolytic bath containing the present ammonium-gold-sulfite complex, which is prepared from the above discussed solution is stable and is operated within the 6-8 pH range. It comprises 1 to 50 g/l of gold metal, 10-400 g/l of ammonium sulfite, 10 to 400 g/l of buffering and conducting salts and it may or may not further comprise alloying metals in the form of water-soluble compounds or complexes.

Thus, this bath which contains the gold in the form of an ammonium-gold-sulfate complex, is able to provide an electrodeposition in acidic media as well as alloyed gold and pure gold, and without needing depolarizers such as arsenic or selenium, even at current densities of 0.25 to 0.5 A./dm², or stabilizers such as polymenars, which was practically impossible with all-alkali-sulfate baths of the present art. Under such conditions, it is possible with this bath to obtain bright and semi-bright 24 karat deposits, homogeneous and non-porous. Further, because of the absence of arsenic, this bath is acceptable in the countries in which the use of this element in industry is restricted or prohibited by laws.

Moreover, this bath being adaptable to acid pH, it is perfectly suited to the deposition of lower karat gold alloys, e.g., 13 to 18 karat. As alloying metals for instance Cd, Pd, Cu, Zn, Sb, In, Ni, Co, Fe, Pb, Bi, Ru, etc., can be mentioned with no limitation. The concentration of these metals in the bath can be e.g. 0.01-150 g/l. These metals are present in the bath as their salts or

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23	US	4379738	A	4									USPAT	
24	US	4377449	A	5									USPAT	
25	US	4377648	A	4									USPAT	
26	US	4310392	A	4									USPAT	
27	US	4238300	A	4									USPAT	
28	US	4212708	A	5									USPAT	
29	US	4192723	A	6									USPAT	

Brief Summary Text - BPTX (8):

Most widely employed gold-plating electrolytes are based on disulphate, ethylenediaminetetraacetate and diaminedisulphate of alkali metals; properties of these gold-plating electrolytes depend on the method of preparing same. This method comprises conversion of aurichlorhydric acid to a sparingly soluble gold compound, auric hydroxide or auric ammoniate to purify from Cl⁻ ions; this gold compound is treated with solutions of sulphates of alkali metals, followed by stabilization of the resulting sulphate complexes in the solution.

Brief Summary Text - BPTX (9):

The thus-produced sparingly soluble gold compounds are low-stable thus causing a partial evolution of metal which, in turn, lowers the desired product yield. This also hinders preparation of highly-concentrated electrolytes for gold-plating which would be stable in storage and use, whereby the efficiency of the gold-plating process is reduced.

Brief Summary Text - BPTX (10):

Known in the art is a gold-plating electrolyte (cf. U.S. Pat. No. 3,533,896) based on potassium disulphate and having the following composition:

Brief Summary Text - BPTX (11):

K.sub.2 [Au(SO₄)₂].sub.2 .sub.2] : 6.8-68

Brief Summary Text - BPTX (17):

This gold-plating electrolyte has a throwing power of up to 90%, yield current up to 70-80%, period of storage at the temperature of 25 degree C. as long as 4 months, starting point of decomposition at D.sub.k above 0.8 A/dm².sup.2.

Brief Summary Text - BPTX (19):

The process for the preparation of this electrolyte comprises treatment of aurichlorhydric acid with a solution of ammonia at a pH of from 3 to 6 to give basic auric ammoniate, followed by decantation of the mother liquor and washing in hot water. The resulting suspension is added to a hot solution of potassium sulphate (to the concentration of 150 g/l), followed by heat-treatment of the resulting mixture at a temperature ranging from 70 degree C. to 90 degree C. for a period of from 48 to 72 hours and addition of an alkali metal salt of ethylenediaminetetraacetic acid, potassium sulphate and potassium hydrogen phosphates. The resulting electrolyte is filtered-off to recover partly precipitated metallic gold. The yield of the thus-produced gold-plating electrolyte is 30 to 60% as calculated for metal gold.

Brief Summary Text - BPTX (20):

GOLD-PLATING ELECTROLYTE

The present invention relates to the preparation of gold compounds and, more specifically, it relates to a gold-plating electrolyte.

FIELD OF THE INVENTION

The present invention is useful in the preparation of gold reagents for electronics, watch making, jewelry and radio-engineering.

BACKGROUND OF THE INVENTION

Known in the art are gold-plating electrolytes based on sodium or potassium dicyanurate and containing 15 to 20% potassium sulphate, potassium hydrogen phosphate, an alkali metal salt of ethylenediaminetetraacetic acid which are prepared by anodic or chemical dissolution of gold in solutions of alkali metal cyanides.

Preparation and use of this type electrolytes are associated with evolution of toxic compounds and the necessity of making them harmless. Said prior art electrolytes make it possible to obtain a good-quality coating only at large thicknesses thereof.

Preparation and use of said prior art gold-plating electrolytes is also accompanied by difficulties encountered in recovery of gold from spent and mother liquors and outgassing of toxic compounds.

Most widely employed gold-plating electrolytes are based on disulphate, ethylenediaminetetraacetate, potassium disulphate and diaminedisulphate of alkali metals; properties of these gold-plating electrolytes depend on the method of preparing same. This method comprises conversion of aurichlorhydric acid to a sparingly soluble gold compound, auric hydroxide or auric ammoniate to purify from Cl⁻ ions; this gold compound is treated with solutions of sulphates of alkali metals, followed by stabilization of the resulting sulphate complexes in the solution.

The thus-produced sparingly soluble gold compounds are low-stable thus causing a partial evolution of metal gold which, in turn, lowers the desired product yield. This also hinders preparation of highly-concentrated electrolytes for gold-plating which would be stable in storage and use, whereby the efficiency of the gold-plating process is reduced.

Known in the art is a gold-plating electrolyte (cf. U.S. Pat. No. 3,533,896) based on potassium disulphate and having the following composition:

K₂[Au(SO₄)₂].sub.2 : 6.8-68

K₂SO₄ : 10-100

K₂HPO₄ : 10-40

water: the balance

(current density is D_k = 0.1-1.2 A/dm²; T = 18°-80° C.)

This gold-plating electrolyte has a throwing power of up to 90%, yield current up to 70-80%, period of storage at the temperature of 25° C. as long as 4 months, starting point of decomposition at D_k above 0.8 A/dm².

This electrolyte ensures the manufacture of high-quality fine coatings with Vickers hardness of from 80 to 220 kg/mm², porosity of from 25 to 30 pores/cm² at the thickness of 15 microns and non-porous at a thickness of below 5 microns.

The process for the preparation of this electrolyte comprises treatment of aurichlorhydric acid with a solution of ammonia at a pH of from 3 to 6 to give basic auric ammoniate, followed by decantation of the mother liquor and washing in hot water. The resulting suspension is added to a hot solution of potassium sulphate (to the concentration of 150 g/l), followed by heat-treatment of the resulting mixture at a temperature ranging from 70 degree C. to 90 degree C. for a period of from 48 to 72 hours and addition of an alkali metal salt of ethylenediaminetetraacetic acid, potassium sulphate and potassium hydrogen phosphates. The resulting electrolyte is filtered-off to recover partly precipitated metallic gold. The yield of the thus-produced gold-plating electrolyte is 30 to 60% as calculated for metal gold.

mother liquor and washing in hot water. The resulting suspension is added to a hot solution of potassium sulphate (to the concentration of 150 g/l), followed by heat-treatment of the resulting mixture at a temperature ranging from 70° to 90° C. for a period of from 48 to 72 hours and addition of an alkali metal salt of ethylenediaminetetraacetic acid, potassium sulphate and potassium hydrogen phosphates. The resulting electrolyte is filtered-off to recover partly precipitated metallic gold. The yield of the thus-produced gold-plating electrolyte is 30 to 60% as calculated for metal gold.

This prior art gold-plating electrolyte has a reduced stability in storage and use, as well as insufficient technological parameters.

SUMMARY OF THE INVENTION

It is the main object of the present invention to provide a gold-plating electrolyte, possessing improved technological parameters and a high stability in storage and use.

This object is accomplished by that a gold-plating electrolyte which contains a complex gold compound with sulphite-ion, an alkali metal salt of ethylenediaminetetraacetic acid, potassium hydrogen phosphate, potassium sulphate and water, in accordance with the present invention contains, as the complex gold compound with sulphite-ion, hexapotassium μ-ethylenediaminetetraacetate bis(gold sulphite) (1/1) with the following proportions of the components (g/l): hexapotassium μ-ethylenediaminetetraacetate bis(gold sulphite) 1/1 10.5-123.0 an alkali metal salt of ethylenediaminetetraacetic acid: 17.8-40.0 potassium sulphate: 12.6-104.0 potassium hydrogen phosphate: 4.7-40.0 water: the balance

Quantitative content of the components in the electrolyte is determined by that at a content of hexapotassium μ-ethylenediaminetetraacetate bis(gold sulphite) below 10.5 g/l, the rate of electrodeposition is insufficient, while at a content thereof above 123 g/l the quality of coatings is impaired. Proportions of the alkali metal salt of ethylenediaminetetraacetic acid, potassium sulphate and potassium hydrogen phosphate affect the quality of coatings, in particular hardness thereof; these proportions are defined by the content of hexapotassium μ-ethylenediaminetetraacetate bis(gold sulphite).

It is advisable that the gold-plating electrolyte according to the present invention contain the components in the following proportions, g/l:

hexapotassium μ-ethylenediaminetetraacetate bis(gold sulphite): 10.5-23.0

disodium-dipotassium salt of ethylenediaminetetraacetic acid: 17.8-40.0

potassium sulphate: 12.6-28.4

potassium hydrogen phosphate: 4.7-10.4

water: the balance

The above-specified amounts of the components of the optimal gold-plating electrolyte composition ensure a maximum reliability thereof in use. Thus, K₂Na₂EDTA in solid amounts inhibits dissociation of ethylenediaminetetraacetate K₄[Au(SO₄)₂EDTA] under the effect of the electric field forces in the layer adjacent to the electrode. In this case the electrochemical reaction proceeds only in a double electric layer and prevents spontaneous decomposition of the complex within the solution volume accompanied by liberation of gold in the powder-like form. K₂SO₄ in the above-

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8. Using the baths of part A, coupons are electroplated as described in Example Three A above, with the following results:

Hardness: 25 = 94.5
Purity: 99.99%
Arsenic: 0.01%

Thallium Modified:
Knoop 25 = 121.5
Gold = 99.99%
Thallium = 0.01%

C. Adjustments to the pH of the unmodified bath are made with tartaric acid and potassium hydroxide, as appropriate, and plating is carried out in a Hull cell for 2 minutes at 0.5 amperes to produce the following results:

pH: 4.5
Brightness: 7.5
Efficiency: 115 mS/amp min

pH: 5.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 6.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 7.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 8.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 9.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 10.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 11.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 12.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 13.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 14.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 15.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 16.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 17.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 18.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 19.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 20.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 21.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 22.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 23.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 24.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 25.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 26.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 27.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 28.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 29.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min

pH: 30.5
Brightness: 115 mS/amp min
Efficiency: 115 mS/amp min</

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L1: (237) (205/266-268).CCL8.

L2: (32763) sulfite or sulfites

L3: (37976) so\$sub.3

L4: (35820) "so.sub.3"

L5: (62544) 12 or 14

L6: (40) 11 and 15

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L11: (253) (205/103-104).CCL8.

L12: (213116) dental or tooth or teeth or crown or crown

L13: (2) L11 and L12

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DB: 115211

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1	P			US 6258239 B1	20010710	12	Process for the manufacture of an electrode for a solid	205/104	205/102;		Stab, Gabriele D. et al.			
2	P			US 4655881 A	19870407		Brush plating method for connector terminals	205/104	205/103;		Tezuka, Junichi et al.			

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☐ Drafts

☐ Pending

☒ Active

☒ L11: (253) (205/103-104). CCL8.

☒ L12: (213116) dental or tooth or teeth or crown or crown

☒ L13: (2) L11 and L12

☒ L14: (31886) electroplating or electrodeposition

☒ L15: (5366) (electrolytics or electrochems) nearl deposi

☒ L16: (34806) L14 or L15

☒ L17: (78) L16 same dental

☒ L18: (4001266) gold or Au

☒ L19: (27) L17 and L18

☒ L20: (381832) pulse or pulses or pulsed or pulsing or pu

☒ L21: (5) L19 and L20

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	U	L	PT	P	Document ID	Issue Date	Pages	Title	Current OR	Current Xref	Retrieval C	Inventor	S	C	3
1	P				US 5782638 A	19980721	20	Ornamental film article for dental substrate decoration	433/206	433/229		Warren, III, A. Daniel et al.			
2	P				US 5316650 A	19940531		Electroforming of metallic glasses for dental	205/67	205/258		Ratzker, Menahem et al.			
3	P				US 4820387 A	19890411		Method and apparatus for making the inner crowns of	205/67	204/229.5; 204/230.7		Yamashita, Atsushi et al.			
4	P				US 4630666 A	19861223		Casting device for dental casts	164/155.2	164/155.4; 164/155.6;		Wismann, Horst			
5	P				US 4528207 A	19850709		Plated dental amalgam alloys	427/2.29	106/1.23; 427/216;		Johnson, Lewis E.			

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Brief Summary Text - BSYX (20):

According to the present invention, it has now been found that such problems can be solved by applying and solidifying a conductive coating material undissolvable in a plating bath on the working model of the tooth on which the state of a tooth is reproduced, even though it is formed of a non-conductive material such as gypsum or synthetic resin, thereby permitting the thus coated portion to be used as the cathode, and substituting a plating for the D.C. plating which has heretofore been used for making an inner crown composed mainly of thick noble metal deposits layer, thereby passing a pulsed current between anode and cathode at a given pulse width and a given pulse current density and in a given duty cycle [(pulse on-time)/(pulse on-time) + (pulse off-time)]. In addition, the problem that only thin metal deposits are obtained on the concave application face can be solved by fixedly immersing an auxiliary electrode formed of the same metal as the metal to be plated in the plating bath between anode and cathode, while its one end is or is not connected to the anode and its other end is opposite and adjacent to a recess in said cathode.

Brief Summary Text - BSYX (21):

More specifically, the present invention provides a method for making an inner crown of a composite-layered crown for the restoration of crowns, said inner crown being composed mainly of a noble metal, wherein an electrically conductive coating material undissolvable in a plating bath is applied and solidified on an inner crown-forming surface of a working model of a tooth on which the state of the tooth to be restored is reproduced and which is formed of a non-conductive material, and electrical connection is made so as to render said conductive coating material cathodic, while said coating material is immersed in said plating bath; an anode is immersed in said plating bath, while said anode is fixedly located on a position opposite to said inner crown-forming position; an auxiliary electrode formed of the same metal as the metal to be plated is immersed in said plating bath between said anode and said cathode, while its one end is or is not connected to said anode and its other end is opposite and adjacent to a recess in said cathode; and a pulsed current is impressed between said anode and said cathode at a pulse width of 0.01 to 50 msec. and a pulsed current density of 1 to 20 A/dm² and in a duty cycle of 1 to 30 % for electroplating, thereby making said inner crown. The present invention also provides an apparatus for carrying out the aforesaid method.

Detailed Description Text - DSYX (2):

Referring to the drawings, there is shown a working model of a tooth 1 on which the state of the tooth to be restored is reproduced and which is formed of an electrically non-conductive material. As the non-conductive material, use may be made of any suitable material capable of easily reproducing the state of a tooth and being removed from an inner crown surface plated on an inner crown-forming surface position and made primarily of a noble metal without dissolving or damaging it such as, for instance, gypsum that is easily dissolvable by a gypsum dissolver manufactured by GC Dental Industrial Corp. and sold under the trade name of "ROCEAN" or synthetic acrylic, epoxy and ethyl silicate resins that are burned out upon heated, leaving behind the inner crown. The metal capable of forming an inner crown by electroplating is mainly a noble metal, and should be a noble metal which can be electroplated and can be securely and hygienically applied under the conditions prevailing in the mouth, such as, for instance, platinum, palladium, rhodium, ruthenium, silver, and silver that may be used alone or in the form of alloys with iron, nickel, copper, cobalt, molybdenum, tungsten and/or indium. It is understood, however,

United States Patent Yamashita et al.

(11) Patent Number: 4,820,387
(43) Date of Patent: Apr. 11, 1989

[54] METHOD AND APPARATUS FOR MAKING THE INNER CROWNS OF COMPOSITE-LAYERED CROWNS FOR RESTORING CROWNS

[75] Inventors: Atsushi Yamashita, Osayama, Ito
Kyotani, Kiamoto, both of Japan
[73] Assignee: GC Dental Industrial Corp., Tokyo,
Japan

[21] Appl. No. 106,463

[22] Filed: Mar. 15, 1988

[30] Foreign Application Priority Data

Apr. 10, 1987 [JP] 62-85761

[51] Int. Cl. C30D 1/06; C30D 17/00

[52] U.S. Cl. 204/4, 204/231

[58] Field of Search 204/4, 231

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Primary Examiner—T. M. Tufatello

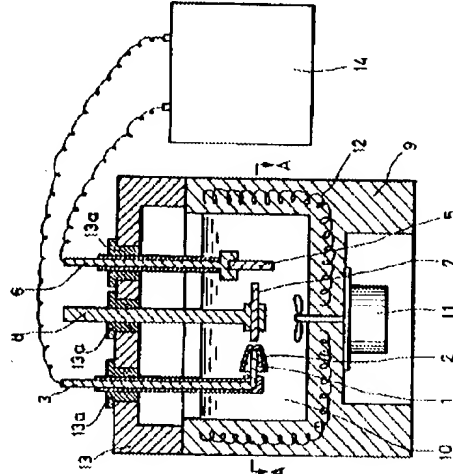
Attorney, Agent, or Firm—Oblom, Fisher, Spivak,

McClelland & Maier

[57] ABSTRACT

In a method and apparatus for making an inner crown of a composite-layered crown for the restoration of crowns, said inner crown being composed mainly of a noble metal, an electrically conductive coating material undissolvable in a plating bath is applied and solidified on an inner crown-forming surface of a working model of a tooth on which the state of the tooth to be restored is reproduced and which is formed of a non-conductive material, and electrical connection is made so as to render said conductive coating material cathodic, while said coating material is immersed in said plating bath; an anode is immersed in said plating bath, while said anode is fixedly located on a position opposite to said inner crown-forming position; an auxiliary electrode formed of the same metal as the metal to be plated is fixedly immersed in said plating bath between said anode and said cathode, while its one end is or is not connected to said anode and its other end is opposite and adjacent to a recess in said cathode; and a pulsed current is impressed between said anode and said cathode at a pulse width of 0.01 to 50 msec. and a pulsed current density of 1 to 20 A/dm² and in a duty cycle of 1 to 30 % for electroplating, thereby making said inner crown.

14 Claims, 1 Drawing Sheet



Document	ID	Pages	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	Kind Codes	Source
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Chung, Kanagawa Shigaku, 22: 32-49, report a metal plate denture electroformed of Ni-CO alloy. In Watanabe et al., Kanagawa Shigaku, 23: 622-628 are reported. Kober et al., Zahnartztl Mitt., 77: 2406-2409. Klet et al. (1987), Dtsch. Zahnartztl Z., 42: 614-617; Klaus, Quintessenz Zahntechnik, 14: 1229-1240 (1988); Menne, Quintessenz Zahntechnik, 15: 1057-1064 (1989); and Stroppe, Dental-Labor (Munch), 38: 201-202 (1990), report work concerning electroforming of metals for dental prostheses. However, all of the alloys studied—nickel, nickel-cobalt—are either not strong enough to be used in thin sections where substantial strength is needed, or they lack biocompatibility.

Brief Summary Text - BSYX (13):

To obtain the objects of this invention, there is provided a process of electroforming metals for dental prostheses, wherein metallic glass alloys of cobalt and phosphorus are electrodeposited on a substrate. Although metallic glass alloys have been known for decades, it was neither known nor at all predictable whether a cobalt-phosphorus glass alloy would be satisfactory for dental prostheses in view of the particular mechanical and corrosion-resistance specifications for such prostheses.

Brief Summary Text - BSYX (33):

The electroforming step is conducted at about 40 degrees -85 degrees C., preferably 75 degrees C., and it is also preferred that an inert gas, such as nitrogen, for example, is purged through the bath at a rate of about 1-10 cc/minute. The current may be direct current, preferably applied at between 150-300 mA/cm² sup. 2 or, alternatively, a pulsed galvanostatic current, preferably at about 300 mA/cm² sup. 2, preferably 200 mA/cm² on and 450 mA/cm² off. Deposition rate can be improved by ultrasonic agitation, rotation or fast stirring of the solution.

Detailed Description Text - DMTX (15):

In order to obtain information on how the Co-P alloys would behave in the mouth, several samples were subjected to accelerated corrosion tests in Hank's solution (Hanks and Wallace, 1949). The only test performed was a simple potentiodynamic scan of the alloys themselves. These were not electrochemically coupled to other metals likely to be in the mouth such as silver amalgams or gold. No attempts were made to optimize the corrosion behavior by alloying. For these measurements, the surface roughness was not controlled. The samples were studied as deposited and were degreased in a series of solvents prior to immersion in the solution. To obtain a comparison with currently used dental alloys, cast Vitallium-RMTM alloy disks were polished with 4000 grit silicon carbide paper. These were then potentiodynamically scanned at the same rate in the same solution. A fresh solution was used for each experiment. The corrosion potential was 200 mV to 300 mV vs. Saturated Calomel Electrode (SCE) for as-deposited Co-P samples and -100 mV to -120 mV vs. SCE for polished Vitallium-RMTM samples. The average corrosion rate (5 samples) was 0.8 mpy for Co-P alloys and 2.3 mpy for the Vitallium-RMTM cast alloy.

Claims Text - CMTX (25):

1. A process according to claim 8, wherein said current is a pulsed

United States Patent [19]

Ratzker et al.

US005316650A

(11) Patent Number: 5,316,650

(45) Date of Patent: May 31, 1994

[54] ELECTROFORMING OF METALLIC GLASSES FOR DENTAL APPLICATIONS

[76] Inventor: Menahem Ratzker, 113 University Blvd., West Silver Spring, Md. 20902, David S. Lashmore, 5506 Woodlyn Rd., Frederick, Md. 21701, John A. Tsek, 13446 Pond Field Ct., Highland, Md. 20777

[21] Appl. No.: 19,489

[22] Filed: Feb. 19, 1993

[51] Int. Cl.⁵ C25D 1/10

[52] U.S. Cl. 205/67, 205/238

[56] Field of Search 205/67, 238

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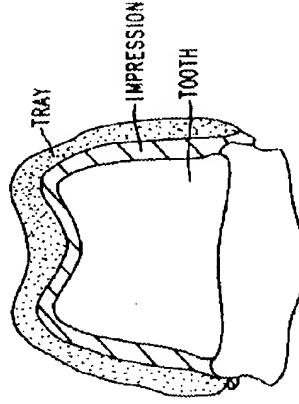
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ABSTRACT

In a process of electroforming a metal to produce a dental prosthesis, the improvement comprising employing as the metal a metallic glass alloy such as cobalt and phosphorus containing 8-50% by weight of phosphorus. The electrolytic bath comprises:

15-300 g/L CoSO₄·7H₂O
40-60 g/L CoCl₂·6H₂O
25-35 g/L H₃PO₃
30-100 g/L H₃PO₄
1-2 ml/L wetting agent
pH about 0.8-2.0

15 Claims, 3 Drawing Sheets



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US-PAT-NO: 4820387

DOCUMENT-IDENTIFIER: US 4820387 A

TITLE: Method and apparatus for making the inner crowns of composite-layered crowns for restoring crowns

----- RWTC -----

Detailed Description Text - DPMX (2):

Referring to the drawings, there is shown a working model of a tooth 1 on which the state of the tooth to be restored is reproduced and which is formed of an electrically non-conductive material. As the non-conductive material, use may be made of any suitable material capable of easily reproducing the state of a tooth and being removed from an inner crown electrolytically on an inner crown-forming surface position and made primarily of a noble metal without dissolving or damaging it such as, for instance, gypsum that is easily dissolvable by a gypsum dissolver manufactured by GC Dental Industrial Corp. and sold under the trade name of "ROCLEAN" or synthetic acrylic, epoxy and ethyl silicate resins that are burned out upon heated, leaving behind the inner crown. The metal capable of forming an inner crown by electrolysis is mainly a noble metal, and should be a noble metal which can be electrolytically and securely and hygienically be applied under the conditions prevailing in the mouth such as, for instance, platinum, palladium, rhodium, ruthenium, gold and silver that may be used alone or in the form of alloys with each other. When the inner crown is formed, it is understood that the inner crown-forming surface of the tooth 1 is built up and fused on the inner crown-forming surface, and a metal is bonded onto the inner crown by the lost-wax casting process, it is required to use a pure metal or its alloy that is unlikely to be softened and deformed at the fusing temperature of porcelain and the casting temperature of the metal. An electrically conductive coating material 2 is applied and solidified on the inner crown-forming surface of the working model of a tooth 1 so as to use it as the cathode. The conductive coating material 2 should have the property of being not dissolved in a plating bath, and may comprise a combination of a binder, viz., cellulose resin, a mixed cellulose/methacrylate/phenolic resin or a mixed methacrylate/polystyrene resin with about 65 to 70 % of silver powders. A cathodic conducting material 3, which is insulated on its outside, is provided so as to fix and retain the working model of a tooth 1 in place and pass a current through the coating material 2 applied and solidified on the surface of the working model of a tooth 1 to render it cathodic. The cathodic conducting material 3 may extend through the working model of a tooth 1 from its side opposite to the inner crown-forming side, and be exposed at its end on that inner crown-forming side to pass a current through the coating material 2, or may be only embedded and fixed in the side of the working model of a tooth 1 which is opposite to the inner crown-forming side. In the latter arrangement, an auxiliary conducting material 4 is needed so as to make electrical connection between end of the coating material 2 opposite to the inner crown-forming side and cathodic conducting material 3. Preferably, the auxiliary conducting material 4 should be insulated on its outer face. When the non-conductive working model of a tooth 1 is formed of gypsum, it is preferred that the working model of a tooth 1 is insulated in the surface area except for the inner crown-forming portion so as to prevent the elution of calcium ions from the gypsum in the plating bath (to be described later), which otherwise results in plating defects. Thus, the provision of such an insulation should preferably be performed.

United States Patent [19] Patent Number: 4,820,387
Yamashita et al. [43] Date of Patent: Apr. 11, 1989

[54] METHOD AND APPARATUS FOR MAKING THE INNER CROWNS OF COMPOSITE-LAYERED CROWNS FOR RESTORING CROWNS

[75] Inventors: Atsushi Yamashita, Okayama; Itao Kyosun, Kinomoto, both of Japan

[73] Assignee: G-C Dental Industrial Corp., Tokyo, Japan

[21] Appl. No: 106,463

[22] Filed: Mar. 18, 1988

[30] Foreign Application Priority Data

Apr. 10, 1987 [JP] Japs 62-85761

[31] Int. Cl. C25D 1/00 C25D 17/00

[32] U.S. Cl. 204/4, 204/231

[36] Field of Search 204/4, 231

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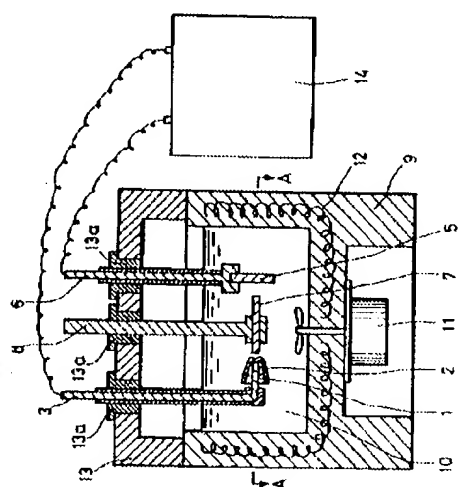
Primary Examiner-T. M. Tufanillo

Attorney, Agent, or Firm-Obiom, Fisher, Spivak, McClelland & Maier

[57] ABSTRACT

In a method and apparatus for making an inner crown of a composite-layered crown for the restoration of a crown, said inner crown being composed mainly of a noble metal, an electrically conductive coating material insoluble in a plating bath is applied and solidified on an inner crown-forming surface of a working model of a tooth on which the state of the tooth to be restored is reproduced and which is formed of a non-conductive material, and electrical connection is made so as to render said conductive coating material cathodic, while said coating material is immersed in said plating bath; an anode is immersed in said plating bath, while said anode is fixedly located on a position opposite to said inner crown-forming position; an auxiliary electrode formed of the same metal as the metal to be plated is fixed for immersion in said plating bath between said anode and said cathode, while its one end is or is not connected to said anode and its other end is opposite and adjacent to a recess in said cathode; and a pulse current is impressed between said anode and said cathode at a pulse width of 0.01 to 50 msec. and a pulsed current density of 1 to 20 A/dm² and in a duty cycle of 1 to 30% for electropositioning, thereby making said inner crown.

14 Claims, 1 Drawing Sheet



FAST - [Default FAST Workspace (Flat Panel LANDSCAPE) - esp. 1]

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49	US 4288298 A	9								USPAT	
50	US 4283839 A	10								USPAT	
51	US 4234666 A	10								USPAT	

Brief Summary Text - BPTX (3):

The invention has particular though non-exclusive application in the construction of dental implants or dental crowns and for explanatory purposes only, is herein described in such context. Thus, in the formation of dental implants, the apparatus may be employed for electrolytically etching a substrate and then electroplating a metal coating onto the substrate, which subsequently is to be coated with a dental porcelain. Such a plating process is proposed for the purpose of providing a bond-aiding interface between a high strength alloy substrate and the porcelain. In the context of electroforming, the apparatus may be employed in the formation of a hollow matrix upon which a dental porcelain is subsequently to be deposited for the purpose of producing a dental crown. However, it is to be understood that the apparatus need not be employed solely for electroplating or electroforming objects for use in dentistry, and that the apparatus may be used in the plating or formation of other objects.

Brief Summary Text - BPTX (5):

Electro-deposition techniques, including both electroplating and electroforming techniques, are in general widely understood and documented. However, to the inventor's knowledge, no previous apparatus has been proposed which is suited to the specialized needs which exist in the construction of dental implants and dental crowns. For such purposes, an electro-depositing apparatus should be capable of predictable performance in electro-depositing metal onto either a small number or a large number of substrates, so that the demands of varying work loads may be met. At the same time, the apparatus should not be so operationally complex as to tax the ability of users who are not specialists in the electro-deposition field. These are seemingly incompatible requirements which, along with other requirements, the present invention seeks to meet.

Detailed Description Text - DETX (2):

The apparatus as shown in the drawings is suitable for use in electroforming hollow metal articles such as gold matrices for dental crowns. In the absence of a suitable electroforming apparatus, such matrices previously have been hand modelled from platinum. In use of the apparatus, a number of gold matrices are electro-formed to a thickness of approximately 200 .mu.m upon male moulds which are formed from artificial stone. Having electroformed the matrices, they are removed from the apparatus, the artificial stone is removed from the matrices and the matrices are covered with an appropriately configured layer of dental porcelain. The invention is not concerned so much with the production of the complete dental crowns, but with the apparatus for use in electroforming matrices for the crowns.

Detailed Description Text - DETX (4):

For the purpose of electroforming gold, the electrolyte may be constituted by a solution having the following composition:

Detailed Description Text - DETX (5):

United States Patent

Rogers

[19]

[11] 4,288,298

[45] Sep. 8, 1981

[54] METHOD AND APPARATUS FOR ELECTROPLATING OR ELECTROFORMING METAL OBJECTS

[76] Inventor: Oliver W. Rogers, 70 Gyrene Bay Rd., Oyster, New South Wales, Australia 2227

[21] Appl. No.: 125,293

[22] Filed: Mar. 11, 1980

[30] Foreign Application Priority Data

Mar. 14, 1979 [AU] Australia PD8025

[51] Int. Cl. C25D 17/06; C25D 17/10

[52] U.S. Cl. C25D 17/06; C25D 17/10

[56] Field of Search 204/228, 204/231, 204/232, 204/233, 204/234, 204/235, 204/236, 204/237, 204/238, 204/239, 204/240, 204/241, 204/242, 204/243, 204/244, 204/245, 204/246, 204/247, 204/248, 204/249, 204/250, 204/251, 204/252, 204/253, 204/254, 204/255, 204/256, 204/257, 204/258, 204/259, 204/260, 204/261, 204/262, 204/263, 204/264, 204/265, 204/266, 204/267, 204/268, 204/269, 204/270, 204/271, 204/272, 204/273, 204/274, 204/275, 204/276, 204/277, 204/278, 204/279, 204/280, 204/281, 204/282, 204/283, 204/284, 204/285, 204/286, 204/287, 204/288, 204/289, 204/290, 204/291, 204/292, 204/293, 204/294, 204/295, 204/296, 204/297, 204/298, 204/299, 204/300, 204/301, 204/302, 204/303, 204/304, 204/305, 204/306, 204/307, 204/308, 204/309, 204/310, 204/311, 204/312, 204/313, 204/314, 204/315, 204/316, 204/317, 204/318, 204/319, 204/320, 204/321, 204/322, 204/323, 204/324, 204/325, 204/326, 204/327, 204/328, 204/329, 204/330, 204/331, 204/332, 204/333, 204/334, 204/335, 204/336, 204/337, 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Abstract Text - ABTX (1):

Method of making artificial tooth inlays and crowns by electrolytically depositing a metal, preferably gold, matrix on a preformed model and subsequently building up porcelain on the matrix. The matrix may then be removed prior to cementing the inlay or crown to the patient's tooth; or alternatively the inlay or crown may be cemented to the patient's tooth together with the matrix.

Inventor Country - INCO (1):

AU

Inventor Group - INSG (1):

Rogers; Olbert William 70 Gynea Bay Road Gynea, New South Wales AU

Priority Country - PROO (1):

AU

Priority Country - PROO (2):

AU

Brief Summary Text - BSTX (2):

It is common practice to construct or crown teeth with metal and/or porcelain. Porcelain has the advantage of being aesthetically more acceptable while metal, commonly, but not necessarily gold or gold alloy, is stronger or at least less brittle.

Brief Summary Text - BSTX (3):

One conventional way of constructing an artificial tooth or crown involves forming a model of a patient's tooth, forming a matrix conforming to the shape of such tooth from e.g. platinum foil by burishing the foil on to the model of such tooth, and thereafter building up and firing porcelain on the foil matrix. Thereafter, the foil is removed from the matrix and the matrix is cemented over the original tooth in the mouth of the patient. Alternatively, a matrix is cast in a metal, e.g. gold alloy, chromium/cobalt or nickel/chromium, and porcelain built up thereon for aesthetic purposes only.

Brief Summary Text - BSTX (6):

According to the present invention a matrix for an artificial tooth construction is formed by electrolytically depositing a suitable metal onto a preformed model and thereafter an artificial tooth construction is built up on the matrix so formed.

United States Patent

Rogers

(19)

3,997,637

Dec. 14, 1976

[54] METHOD OF MAKING TOOTH RECONSTRUCTIONS SUCH AS INLAIS AND CROWNS

[76] Inventor: Olbert William Rogers, 70 Gynea Bay Road, Gynea, New South Wales, Australia

[22] Filed: Apr. 23, 1975

[21] Appl. No.: 570,740

[30] Foreign Application Priority Data

Apr. 29, 1974 Australia 738774
Dec. 31, 1974 Australia 150774

[52] U.S. Cl. 204/30, 264/22, 264/46, 264/129, 264/221

[31] Int. Cl. A61C 13/08, A61C 5/10

[58] Field of Search 264/16, 17, 21, 19, 264/219, 221, 223, 226, 227, 265, 317, 238, 332, 18, 56, 129, 322, 12, 13, 8, 204/20, 30

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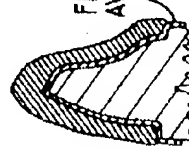
Anon., Technique of Fusing Porcelain to Gold, Julius Adler, Inc., N.Y., (1939), pp. 2-15 relied on.

Primary Examiner—Willard E. Hoag
Attorney, Agent, or Firm—Hall, Gross, Simpson, Van Santen, Steadman, Chiara & Simpson

[57] ABSTRACT

Method of making artificial tooth inlays and crowns by electrolytically depositing a metal, preferably gold, matrix on a preformed model and subsequently building up porcelain on the matrix. The matrix may then be removed prior to cementing the inlay or crown to the patient's tooth, or alternatively the inlay or crown may be cemented to the patient's tooth together with the matrix.

5 Claims, 43 Drawing Figures



FREE MARGIN OF SOFT GOLD
AVAILABLE FOR ADAPTATION

MATRIX LEFT IN SITU-PORCELAIN
JACKET CROWN PLACED ON TOOTH

EAST - [Default EAST Workspace (Flat Panel LANDSCAPE) wsp1]

[illegible]

4/2003

FAST - Drafts FAST Workspace (Flat Panel LANDS/CAPE) wsp 11

File View Edit Lock Window Help



Drafts

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L1: (253) (205/103-104).CCLS.

L2: (30714) duty adj cycle

L3: (31) 11 and 12

L4: (4001266) gold or Au

L5: (13) 13 and 14

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US-PAT-NO: 466567

DOCUMENT-IDENTIFIER: US 466567 A

TITLE: Automated alternating polarity pulse electrolytic processing of electrically conductive substances

----- KWIE -----

Brief Summary Text - BSTR (5):

Moreover, it is economically advantageous when electroplating precious metals such as gold, silver, rhodium, etc., to utilize very dilute solutions. This achieves minimum drag out losses and reduces precious metal inventory cost in the solutions. Drag out losses are the plating metal losses which occur as the part being plated is removed from the plating solution. It should be borne in mind, however, that very dilute solutions undergo a reduction in conductivity during plating as the metal ion concentration diminishes. Consequently, the electroplating voltage must increase to maintain a constant current density on the parts being plated. However, as alluded to above, the plating voltage must be maintained below the burn voltage at which the metal deposit becomes unacceptably dull, rough and nodular. Moreover, electroplating at or above the burn voltage results in excessive amounts of molecular hydrogen being infused into the part being plated rendering it imbricated. In some cases, the part being plated may become etched. However, if the metal concentration in the solution is increased to remedy these effects, it causes the uniformity of the deposit thickness to decrease.

Detailed Description Text - DEXT (44):

Also connected to the collector of Q2 through series resistor R11 are back-to-back light emitting diodes LED1 and LED2 which are connected to ground. Light emitting diodes LED1 and LED2 are activated depending on the polarity at terminal X to indicate the same during the electrolytic process of the output signal. Terminal Y is connected to the -15 volt terminal on the microprocessor power supply 32.

Current US Cross Reference Classification - CCCR (3):

4,666,567

excessive reverse pulse durations would produce dissolution of the material plated on the part and a dull coating appearance. A simultaneous reduction in the forward pulse duration after a small increase in reverse pulse duration will permit a continuing decrease in the forward/reverse pulse duration ratio without having reverse pulses of an excessive length.

PROCESS DESCRIPTION

A comparison of a prior art anodizing current waveform with the waveform of the present invention is illustrated in FIGS. 2A and 2B. FIG. 2A shows a variable polarity waveform as disclosed in U.S. Pat. No. 3,983,014. While not directly analogous to the plating process of the present invention, the anodizing current disclosed in U.S. Pat. No. 3,983,014 may assist in obtaining a better understanding of the present invention. According to the prior art, a forward pulse of a fixed duration is followed by a slightly shorter reverse pulse of a fixed duration in a periodic manner until either the desired coating thickness is reached or the coating breakdown voltage is reached. It should be understood that the duration of one complete cycle in the FIG. 2A waveform is typically one-tenth of one second or less as opposed to waveform durations of one microsecond or more in the FIG. 2B embodiment for the plating cycle. It should also be noted that FIG. 2B does not show zero current periods between positive and negative current pulses, although such finite periods actually exist. The zero current periods are omitted in the figure to denote a relatively short off-time between pulses compared to the pulse durations themselves.

The present invention is characterized by two separate cycles—the conditioning cycle and a plating cycle. The conditioning cycle, which may last 10 minutes, comprises short and constant duration pulses of both forward and reverse current polarity which "condition" the substrate in order to improve the deposit adhesion to it. The plating cycle shown in FIG. 2B is divided into two phases, phases a and b. Each of these phases includes a reduction in the time ratio of forward to reverse power applied. During phase a, a decreasing duration forward pulse is interrupted by an increasing duration negative pulse. In phase b, if further plating is necessary, in order to build up an extremely thick coating or the solution is to be exhausted of plating ions, the pulse durations of forward and reverse pulses are maintained constant and the amplitude of the forward current pulses is decreased. In many instances, phase b is unnecessary because a sufficiently thick coating can be built up during phase a. However, it should be understood that phase b can advantageously be included with phase a when necessary. Furthermore, as long as the time ratio of forward to reverse power is decreasing as a function of plating ion depletion, the precise phase utilized is not of critical importance. As should now be clear to the artisan, the magnitude of the reverse current flow could be increased while the forward current flow is constant or decreased in order to decrease the forward to reverse power ratio. Furthermore, combinations of phases a or b can be altered to provide a greater or quicker decrease in the forward to reverse power ratio.

In the embodiment of FIG. 2B, at the beginning of phase a, a maximum forward pulse may be applied for about 30 milliseconds followed by a reverse pulse applied for about 0.5 milliseconds. As the concentration of plating metal ions decreases, the process voltage in-

creases to maintain a constant current input. In order to control the process voltage, the forward and reverse pulse duration ratios change in accordance with, for example, microprocessor instructions or the like, which are dependent upon the instantaneous process voltage V(t) necessary to maintain the appropriate current level. Toward the end of the plating cycle, when the coating has attained its maximum thickness, the final current waveform is such that the forward pulse duration is at a minimum value and the reverse pulse duration is at a maximum value. For example at the end of phase a, the forward pulse duration may be on the order of 5 milliseconds and the reverse pulse duration of 5 milliseconds. The maximum forward pulse duration and maximum reverse pulse duration illustrated are not drawn to scale and reflect only relative changes in pulse durations of forward and reverse pulses.

As noted earlier, in many instances, the maximum coating thickness is not desired, and in the preferred embodiment, the complete waveform alteration pattern shown in FIG. 2B is not required nor is maximum depositing ion concentration depletion required, and in the preferred embodiment, the complete waveform alteration pattern shown in FIG. 2B is not required in order to control the process voltage. The process voltage to the microprocessor control is illustrated in FIG. 1B where V_a is the process voltage applied between the part and the anode at any point in time. It should be understood that the excursion or variation of V_a shown in FIG. 1B is purely illustrative and intended only to depict the general automated voltage trend of a preferred embodiment. Voltage limits V₁ and V₂ are independently adjustable to control the process voltage excursions, thereby controlling the coating thickness and properties as well as the degree of plating ion concentration in the electroplating solution. V₁ is defined as the process voltage at which the microprocessor begins to alter the initially applied waveform by decreasing the forward to reverse power ratio. V₂ is the process voltage at which the microprocessor would generate a final waveform configuration which would have a forward pulse minimum duration and a reverse pulse maximum duration. A reduction of plating current density could be utilized if the process voltage tended to go above V₂ by means of a suitable voltage limiting constant current controlled forward power supply.

A functional block diagram of one embodiment of the present invention is shown in FIG. 3 which includes a standard plating tank containing an ambient temperature or heated electroplating solution 12. The solution 12 may be air or mechanically agitated. A solution inlet anode or an anode of the metal being deposited 14 is provided in the tank to complete the current flow path to the part 16 which is to be plated. Where soluble anodes are used during operation of the apparatus, particles may flake off the anode. Therefore, a loose bag 17 of material unaffected by the electroplating solution, such as "Dynal" can be arranged around the anode to retain any particles. The anode 14 and the part 16 to be plated 16 are connected to a power driver 18 which is supplied by positive (plating) and negative (diffusion) power supplies 20 and 22 respectively. An analog to digital converter 24 samples the voltage applied between the anode and the part to be plated and provides an input to the waveform generator and controller 26. The controller may include a visual display 28 and

4/2003

The screenshot displays the EASI - (Default) FAS1 Workspace interface. At the top, there's a title bar indicating the workspace name and version. Below it is a standard menu bar with options like File, View, Edit, Tools, Window, and Help. A toolbar with various icons follows. The main area is split into two panes. The left pane lists several draft documents, including 'L1: (516) (204/272).CCLS.', 'L2: (31937) electroplat\$ or electrodeposit\$', and 'L3: (89) 11 and 12'. The right pane provides a detailed view of the selected document, showing metadata such as 'Title', 'Document ID', 'Issue Date', 'Pages', 'Current OR', 'Current XRef', 'Retrieval C', 'Inventor', and a series of alphanumeric codes ('S', 'C', 'Z', '3', '3'). The bottom of the window features a status bar that reads 'Ready'.

Trevelyan
 Walter L. Chubbard, Jr.
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Document ID	Pages	U	S	G	P	Kind Codes	Source
US 0902892 A	4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>		USOCP
218 US 0899827 A	3	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>		USOCP
219 US 0897291 A	3	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>		USOCP
220 US 0891361 A	8	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>		USOCP
221 US 0884075 A	2	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>		USOCP
222 US 0883756 A	5	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>		USOCP
223 US 0881010 A	5	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>		USOCP

US-PAT-NO: 0902892

DOCUMENT-IDENTIFIER: US 0902892 A

TITLE: OCR SCANNED DOCUMENT

DATE-ISSUED: November 3, 1906

INVENTOR: Name not available

US-CL-CURRENT: 205/151, 204/229.4, 204/272

NOOCRTEXT

BEST AVAILABLE COPY

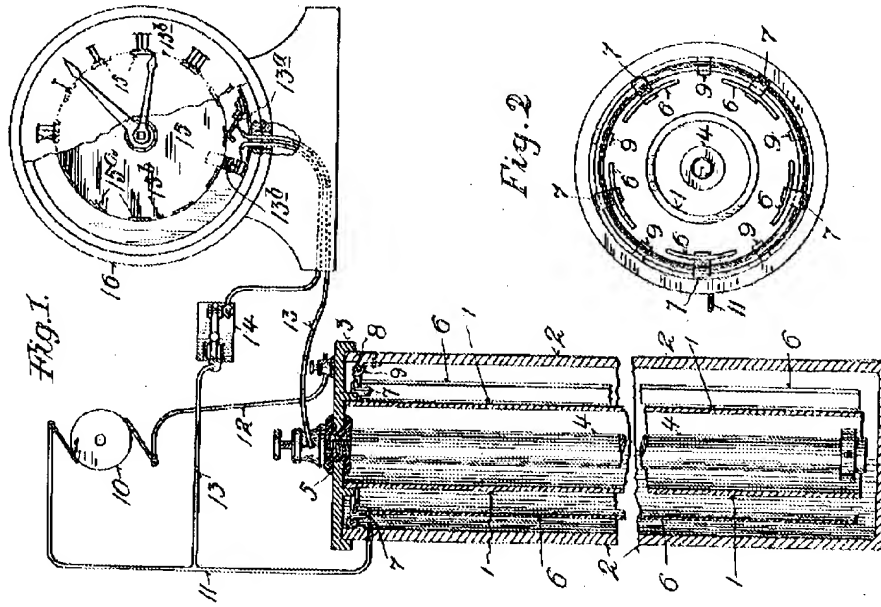
G. A. LUTZ.

METHOD OF AND APPARATUS FOR ELECTROPLATING.

APPLICATION FILED JUNE 27, 1908.

902,892.

Patented Nov. 3, 1908.



Witnesses:
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Inventor
 Geo. A. Lutz
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 J. F. Brown

Document ID#	Pages	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	251	252	253	254	255	256	257	258	259	260	261	262	263	264	265	266	267	268	269	270	271	272	273	274	275	276	277	278	279	280	281	282	283	284	285	286	287	288	289	290	291	292	293	294	295	296	297	298	299	300	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334	335	336	337	338	339	340	341	342	343	344	345	346	347	348	349	350	351	352	353	354	355	356	357	358	359	360	361	362	363	364	365	366	367	368	369	370	371	372	373	374	375	376	377	378	379	380	381	382	383	384	385	386	387	388	389	390	391	392	393	394	395	396	397	398	399	400	401	402	403	404	405	406	407	408	409	410	411	412	413	414	415	416	417	418	419	420	421	422	423	424	425	426	427	428	429	430	431	432	433	434	435	436	437	438	439	440	441	442	443	444	445	446	447	448	449	450	451	452	453	454	455	456	457	458	459	460	461	462	463	464	465	466	467	468	469	470	471	472	473	474	475	476	477	478	479	480	481	482	483	484	485	486	487	488	489	490	491	492	493	494	495	496	497	498	499	500	501	502	503	504	505	506	507	508	509	510	511	512	513	514	515	516	517	518	519	520	521	522	523	524	525	526	527	528	529	530	531	532	533	534	535	536	537	538	539	540	541	542	543	544	545	546	547	548	549	550	551	552	553	554	555	556	557	558	559	560	561	562	563	564	565	566	567	568	569	570	571	572	573	574	575	576	577	578	579	580	581	582	583	584	585	586	587	588	589	590	591	592	593	594	595	596	597	598	599	600	601	602	603	604	605	606	607	608	609	610	611	612	613	614	615	616	617	618	619	620	621	622	623	624	625	626	627	628	629	630	631	632	633	634	635	636	637	638	639	640	641	642	643	644	645	646	647	648	649	650	651	652	653	654	655	656	657	658	659	660	661	662	663	664	665	666	667	668	669	670	671	672	673	674	675	676	677	678	679	680	681	682	683	684	685	686	687	688	689	690	691	692	693	694	695	696	697	698	699	700	701	702	703	704	705	706	707	708	709	710	711	712	713	714	715	716	717	718	719	720	721	722	723	724	725	726	727	728	729	730	731	732	733	734	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760	761	762	763	764	765	766	767	768	769	770	771	772	773	774	775	776	777	778	779	780	781	782	783	784	785	786	787	788	789	790	791	792	793	794	795	796	797	798	799	800	801	802	803	804	805	806	807	808	809	810	811	812	813	814	815	816	817	818	819	820	821	822	823	824	825	826	827	828	829	830	831	832	833	834	835	836	837	838	839	840	841	842	843	844	845	846	847	848	849	850	851	852	853	854	855	856	857	858	859	860	861	862	863	864	865	866	867	868	869	870	871	872	873	874	875	876	877	878	879	880	881	882	883	884	885	886	887	888	889	890	891	892	893	894	895	896	897	898	899	900	901	902	903	904	905	906	907	908	909	910	911	912	913	914	915	916	917	918	919	920	921	922	923	924	925	926	927	928	929	930	931	932	933	934	935	936	937	938	939	940	941	942	943	944	945	946	947	948	949	950	951	952	953	954	955	956	957	958	959	960	961	962	963	964	965	966	967	968	969	970	971	972	973	974	975	976	977	978	979	980	981	982	983	984	985	986	987	988	989	990	991	992	993	994	995	996	997	998	999	1000	1001	1002	1003	1004	1005	1006	1007	1008	1009	1010	1011	1012	1013	1014	1015	1016	1017	1018	1019	1020	1021	1022	1023	1024	1025	1026	1027	1028	1029	1030	1031	1032	1033	1034	1035	1036	1037	1038	1039	1040	1041	1042	1043	1044	1045	1046	1047	1048	1049	1050	1051	1052	1053	1054	1055	1056	1057	1058	1059	1060	1061	1062	1063	1064	1065	1066	1067	1068	1069	1070	1071	1072	1073	1074	1075	1076	1077	1078	1079	1080	1081	1082	1083	1084	1085	1086	1087	1088	1089	1090	1091	1092	1093	1094	1095	1096	1097	1098	1099	1100	1101	1102	1103	1104	1105	1106	1107	1108	1109	1110	1111	1112	1113	1114	1115	1116	1117	1118	1119	1120	1121	1122	1123	1124	1125	1126	1127	1128	1129	1130	1131	1132	1133	1134	1135	1136	1137	1138	1139	1140	1141	1142	1143	1144	1145	1146	1147	1148	1149	1150	1151	1152	1153	1154	1155	1156	1157	1158	1159	1160	1161	1162	1163	1164	1165	1166	1167	1168	1169	1170	1171	1172	1173	1174	1175	1176	1177	1178	1179	1180	1181	1182	1183	1184	1185	1186	1187	1188	1189	1190	1191	1192	1193	1194	1195	1196	1197	1198	1199	1200	1201	1202	1203	1204	1205	1206	1207	1208	1209	1210	1211	1212	1213	1214	1215	1216	1217	1218	1219	12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[illegible]This image shows a blank page from a document. There are faint vertical lines along the left edge, possibly indicating binding or scanning artifacts. A small dark mark is visible at the bottom right corner. The rest of the page is white and contains no text or other markings.

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FAST - (Default FAST Workspace (Flat Panel LAMOGRAPH) vwp1)

File View Edit Tools Window Help

Drafts
☐ Pending
☒ Active

- ☒ L1: (31886) electroplating or electrodeposits
- ☒ L2: (5354) (electrolytics or electrochemicals) nearl deposits
- ☒ L3: (34795) 11 or 12
- ☒ L4: (213116) dental or tooth or teeth or crown or crowns
- ☒ L5: (221) 13 same 14
- ☒ L6: (4001266) gold or Au
- ☒ L7: (388003) pulse or pulses or pulsed or pulsing or pulsation or pulsating
- ☒ L8: (67) 15 and 16
- ☒ L9: (9) 18 and 17
- ☒ L10: (58) 18 not 19
- ☒ L11: (237) (205/266-268). CCLB.
- ☒ L12: (82359) (current or voltage) near3 17
- ☒ L13: (990) (plate or plates or plated or plating) near1 17
- ☒ L14: (82980) 112 or 113
- ☒ L15: (10) 111 and 114

- ☒ failed
- ☒ saved
- ☒ favorites
- ☒ tagged (0)
- ☒ UDC
- ☒ Queue
- ☒ Trash

DB:

Default operator: OR

111 and 114

☐ Print

☐ History of items added

U	I	PI	P	Document ID	Issue Date	Pages	Title	Current OR	Current Xref	Retrieval C	Inventor	S	C	2	3
1	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6203936 B1	20010320	7	Lightweight metal bipolar plates and methods for Electrochemical Cells	429/44	204/290.03; 204/290.06; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	204/290.03; 204/290.06; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Cisar, Alan J. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
2	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5863400 A	19990126		Coated Article	428/627	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Drummond, Humphrey John Jardine et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
3	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5783313 A	19980721		Low stress electrodeposition of gold for X-ray mask	205/261; 205/266; 205/266; 430/5	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Moysen, III, Stephen R. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5318607 A	19940607		Method of and apparatus for plating using controlled tartaric acid and carbonate salts	205/261; 205/266; 205/266; 430/5	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Daukscher, William J. et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
5	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5266183 A	19931130		Method of and apparatus for plating using controlled tartaric acid and carbonate salts	205/261; 205/266; 205/266; 430/5	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Macdonald, David I.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
6	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 4537663 A	19850827		Gold plating bath containing gold plating bath and method using maleic anhydride	205/261; 205/266; 205/266; 430/5	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Fletcher, Augustus et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
7	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 4465564 A	19840814		Gold plating bath containing gold plating bath and method using maleic anhydride	205/261; 205/266; 205/266; 430/5	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Fletcher, Augustus et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
8	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 4396471 A	19830802		Composition and method for gold plating	205/261; 205/266; 205/266; 430/5	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Fletcher, Augustus et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
9	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 4253920 A	19810303		Method of electroplating precious metals in localized	205/261; 205/266; 205/266; 430/5	204/290.03; 204/192.17; 204/403.11; 205/261; 205/266; 205/266; 430/5	Koonitz, Donald Eldridge et al.	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
10	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 4001093 A	19770104							<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

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US-PAT-NO:

4465564

DOCUMENT-IDENTIFIER:

US 4465564 A

TITLE: Gold plating bath containing tartrate and carbonate salts

----- KWIC -----

Brief Summary Text - BSYX (7):

Despite the foregoing, a demand exists for a gold plating bath that is capable of producing electrodeposits of high purity, and particularly one that is capable of producing deposits of sufficient purity to meet the requirements of the semiconductor industry, using either continuous or batch electrodeposition. A need also exists for such a bath of relatively low specific gravity, which is capable of operating under high current density plating conditions at relatively low gold concentrations, and which has a high tolerance to contamination, such as from copper. Finally, there is a demand for a bath having the foregoing features and advantages, from which gold can be alloyed with various metals to produce a variety of characteristics in the deposit, and to extend the range of brightness exhibited thereby.

Brief Summary Text - BSYX (23):

Various types of plating apparatus may be employed in connection with the compositions and methods of the present invention, including equipment for barrel and rack plating, and for high speed continuous selective plating. In addition to steady direct current plating, pulse plating can be employed to produce good quality, non-porous deposits at relatively high speeds with the least amount of gold content.

Detailed Description Text - DETX (27):

The uncontaminated bath of Example Three above is tested for use in producing a semi-conductor package in which a silicon chip is bonded by heat and pressure to a gold electroplated surface, and the leads are electrically connected from the gold plated surface to the silicon chip by thermosonic or ultrasonic means. In the test, the surface for bonding is first plated with nickel from a sulfamate solution to a thickness of 1.25 micrometers, and is then plated in the gold bath to produce a 1.5 micrometer deposit, using either pulsed or non-pulsed DC current (average current density of 3 ASF). The resultant packages are subjected to standard wire bond test procedures used in the semiconductor industry, involving evaluation of the assemblies in the "as plated" condition as well as after heat cycling at different temperatures and for varying periods, and satisfy all applicable criteria.

Claims Text - CMTX (11):

9. The method of claim 7 wherein said potential is applied by pulsed direct current.

United States Patent

Fletcher et al.

[19]

[31] Patent Number: 4,465,564

[45] Date of Patent: Aug. 14, 1984

[54] GOLD PLATING BATH CONTAINING TARTRATE AND CARBONATE SALTS

[75] Inventors: Augustus Fletcher, Bristol, Conn.; David N. Cockayne, Farling, Hong Kong; William L. Moriarty, South Meriden, Conn.

[73] Assignee: American Chemical & Refining Company, Inc., Waterbury, Conn.

[21] Appl. No.: 607,763

[22] Filed: Jun. 27, 1983

[31] Int. Cl.: C25D 3/48; C25D 3/62

[52] U.S. Cl.: 204/44; 204/43 G; 204/DIG. 9

[54] Field of Search: 204/43 G, 46 G, 109, 204/123, 44, DIG. 9

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Primary Examiner—G. L. Kaplan

[57] ABSTRACT

A bath for electrodepositing gold utilizes, as the essential ingredients of its electrolyte, salts providing the tartrate radical and the carbonate radical. The bath will have a pH value ranging from approximately neutral to highly alkaline, and adjustment to lower the pH will most advantageously be made utilizing tartaric acid. The bath operates with relatively low gold concentrations, and is capable of producing highly pure deposits and highly desirable alloy deposits. It is especially well suited for semi-conductor industry applications.

15 Claims. No Drawing